Final Report on
Task Order 12

THE TOTAL NORMAL ABSORPTANCE OF A LAMPBLACK COATING ON COPPER

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Contract NAS8-5196

Southern Research Institute
Birmingham, Alabama
April 2, 1965
7240-1481-12-XXXIV

TABLE OF CONTENTS

SCOPE AND SUMMARY

This is the final report on Task Order 12 of Contract No. NAS8-5196 for National Aeronautics and Space Administration. This task order concerned the determination of the total normal absorptance of a thin lampblack coating on a smooth, clean, copper surface over the temperature range from 70°F to 1500°F.

The absorptance was determined by measuring the emittance of the coating and assuming emittance equal to absorptance. This assumption was confirmed by Kirchoff's law since the data obtained indicated that the lampblack coating radiated as a gray body.

The emittance was measured by comparing the energy received by a radiometer from the sample to that received from a blackbody at the same temperature. The lampblack coating exhibited a total normal emittance that remained constant at 0.90 over the entire temperature range.

SPECIMEN MATERIAL AND PREPARATION

The lampblack employed for this evaluation was supplied by NASA and was designated as Lamp Black 1's prepared by the L. Martin Company, Tacony, Pennsylvania.

The lampblack was mixed with carbon tetrachloride and painted on the copper surface, resulting in a thin (less than 0.001 inch thick) coating of lampblack after evaporation of the carbon tetrachloride. The copper substrate had a smooth surface finish that was prepared by hand buffing with "Grade 2"[1] steel wool. To avoid handling prior to the runs, the coating was applied after the copper substrate was installed and instrumented within the apparatus.

---

1. American Flex-Fold Steel Wool

APPARATUS AND PROCEDURE

The total normal emittance was measured by comparing the energy received from the sample to that received from a blackbody cavity maintained at the same temperature. Two arrangements of the same basic apparatus were used to cover the entire temperature range of 70°F to 1500°F. The standard emittance apparatus that has been employed here for the past several years was utilized for the evaluation from 600°F to 1500°F. For the lower range of 70°F to 800°F a modification of the standard apparatus was employed. The standard apparatus and procedure are described briefly in the text and in detail in the Appendix; the special procedures employed are described in detail in the following sections.

Standard Apparatus Used from 600°F to 1500°F

The temperature of the coated surface was measured with a chromel-alumel thermocouple spot welded to the viewed surface of the copper disc. The thermocouple wires of .005 inch diameter were extended directly from the surface and brought through a double bore alumina tube situated in the mid-plane of the disc, as shown in Figure 1, to provide thermal guarding along the initial length of the wire.

The calibration of the radiometer was checked with a small blackbody cavity of 2:1 aspect ratio inserted in the field of the flat coil. From 500°F to 1500°F the radiometer output during this calibration was slightly higher than the previous calibration (shown in the Appendix), so the recalibration curve, shown in Figure 2, was used for the computation of emittance.

The emittance was calculated by dividing the radiometer output when viewing the specimen by the output indicated on the calibration curve of Figure 2 for the same temperature.

Modified Apparatus Used from 70°F to 800°F

The total normal emittance from 70°F to 800°F was determined by employing a modified apparatus which incorporated the basic parts of the standard apparatus. The modifications were made in order to allow sufficient radiant exchange between the radiometer and the low temperature specimen, and minimize extraneous radiation from the surroundings, thus providing the maximum millivolt output and minimum background "noise" from the radiometer.

The apparatus is schematically shown in Figure 3. The blackbody cavity of approximately 4:1 aspect ratio was fabricated from copper to the configuration shown in Figure 4, and was heated resistively with Kanthal wire windings encased within the surrounding casting of alumina cement. The cavity walls were darkened by oxidizing the copper and applying a uniform coating of lampblack. The two specimens also had separate heaters and with the blackbody cavity were installed on the transite support bar which in turn was clamped to the movable blank off valve. This enabled the specimens and the blackbody to be rapidly aligned in view of the radiometer. The alignment was preset and maintained during the run by external stops.

The temperatures of the specimens were monitored by chromel-alumel thermocouples welded to the copper substrate of the viewed surface. Thermal guarding of the 0.005 inch diameter thermocouple wires was accomplished by inserting the wires in a double bore alumina tube which ran diagonally through the corner of the heater block as illustrated in Figure 3. The temperature of the blackbody was monitored with two chromel-alumel thermocouples inserted at different depths within the wall of the cavity.

The radiometer housing was designed to cool and maintain the radiometer at an even temperature and also to position the radiometer near the specimen. As shown in Figure 3, copper cooling coils were soldered to the housing and, depending on the desired specimen temperature, liquid nitrogen or water was employed for cooling. For specimen temperatures from 70°F to 200°F, liquid nitrogen was used; above 200°F water was employed. The entire apparatus was insulated with Fiberfrax and other insulating materials.

The evaluation was performed in a dry helium atmosphere to eliminate frosting and oxidation. This atmosphere was obtained by initially evacuating the system several times, refilling with dry helium and maintaining a helium purge throughout the run.

After providing the dry helium atmosphere, liquid nitrogen was circulated through the cooling coils and the radiometer and housing were allowed to reach steady state. For the cases where water cooling was used, the water was circulated through the coils several hours previous to the run and the helium atmosphere was provided when the radiometer reached a steady temperature. For both cases, data were not obtained until the radiometer zero reading (obtained with the radiometer viewing the blank off valve) remained steady, indicating thermal equilibrium throughout the thermopile. The data were obtained by (1) recording the temperature of the specimen or blackbody, (2) obtaining a radiometer zero reading, (3) aligning the specimens or blackbody cavity in view, (4) obtaining the radiometer output, and (5) recording the final temperature of the specimen or blackbody.

The blackbody cavity was utilized as a reference during each run because the radiometer zero and output readings between runs were slightly affected by some variation in the final temperature of the radiometer after cool-down. For both cases employing either the liquid nitrogen or water cooling, several readings were taken viewing the specimens and the blackbody at various temperatures. The data of radiometer output versus temperature for the blackbody radiation are shown in Figures 5 and 6 for the runs with liquid nitrogen and water cooling, respectively. The emittance of the specimens was calculated by dividing the radiometer millivolt output while viewing the specimen by the output indicated on the curves of Figures 5 and 6 at the same temperature.

During the runs with liquid nitrogen cooling, the temperature of the specimens varied slightly while each point was being monitored. This was caused by the small helium purge which impinged on the specimen while it was in the field of view of the radiometer. However, the blackbody temperatures before and after the radiometer reading were not affected significantly and the temperature gradients along the axis of the cavity remained at less than 5°F. With water cooling, the temperature drop of the specimens while monitoring the reading was less than 2°F and gradients within the blackbody cavity were negligible. In calculating the emittance, the average temperature was used.

DATA AND RESULTS

The total normal emittance of the lampblack coating determined from 70°F to 1500°F is shown in Figure 7 and Table 1. The emittance remained at a constant value of 0.90 over the entire temperature range.

The smooth curve was fitted to the data favoring the higher values obtained with the modified apparatus in the temperature range from 600°F to 800°F since the modified apparatus had better response and was considered more accurate than the standard arrangement at these temperatures.

This value of 0.90 is slightly lower than the values of 0.92 to 0.96 reported in the literature. This was attributed to the minimum thickness of the coating and the relatively low emittance of the substrate.

With review of the above data, the assumption of emittance being equal to absorptance for this case was considered valid. Under Kirchoff's law, the total emittance will equal total absorptance provided the surface radiates as a gray body. The constant value of emittance over the entire temperature range indicated gray body radiation at least over the corresponding wavelength band of 2.6 to 10 microns. Gray body radiation probably occurs over a wider wavelength range since the dull black appearance of the surface also qualitatively suggests that the absorptance is close to 0.9 at the lower wavelengths in the visible range.

Submitted by:

W. T. Engelke

W. T. Engelke
Associate Engineer
Thermodynamics Section

Approved by:

C D Pears

C. D. Pears, Head
Mechanical Engineering Division

7240-1481-12-XXXIV
(10:20) lw

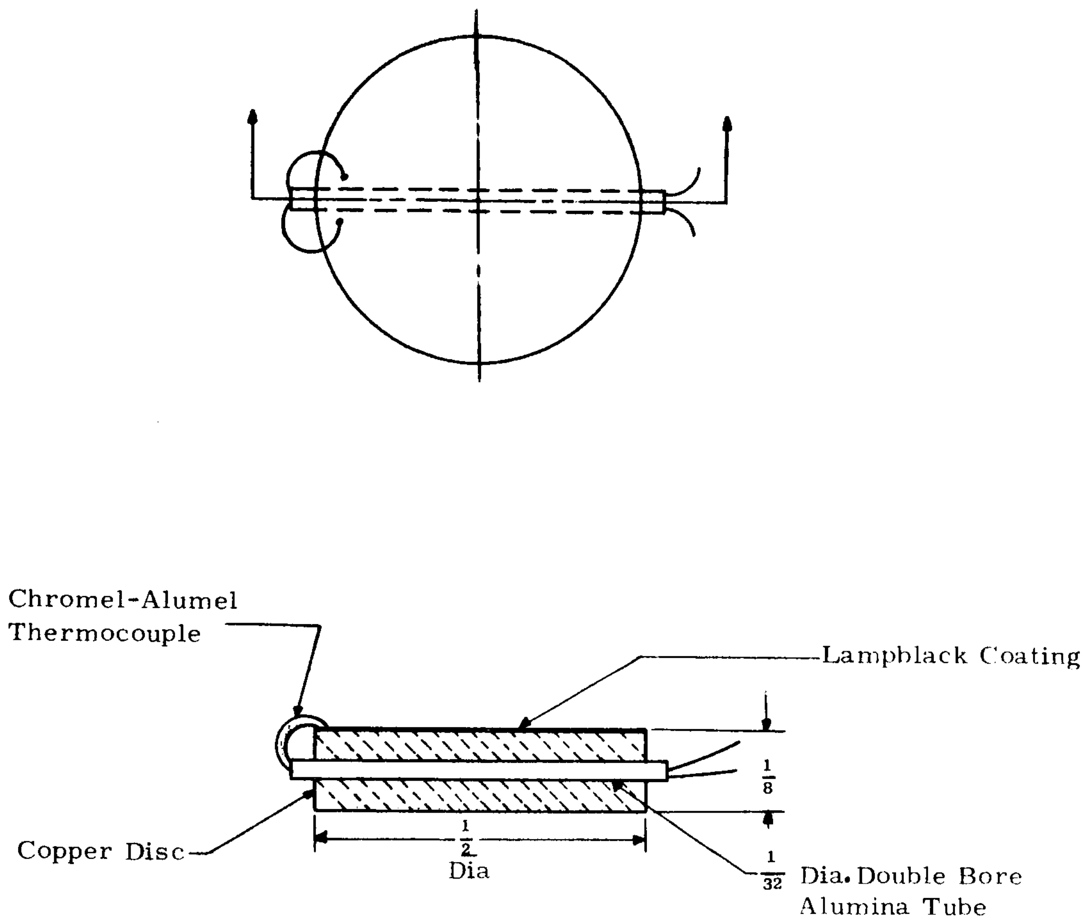


Figure 1. Specimen Configuration Used in the Standard Apparatus for Temperatures from 600°F to 1500°F

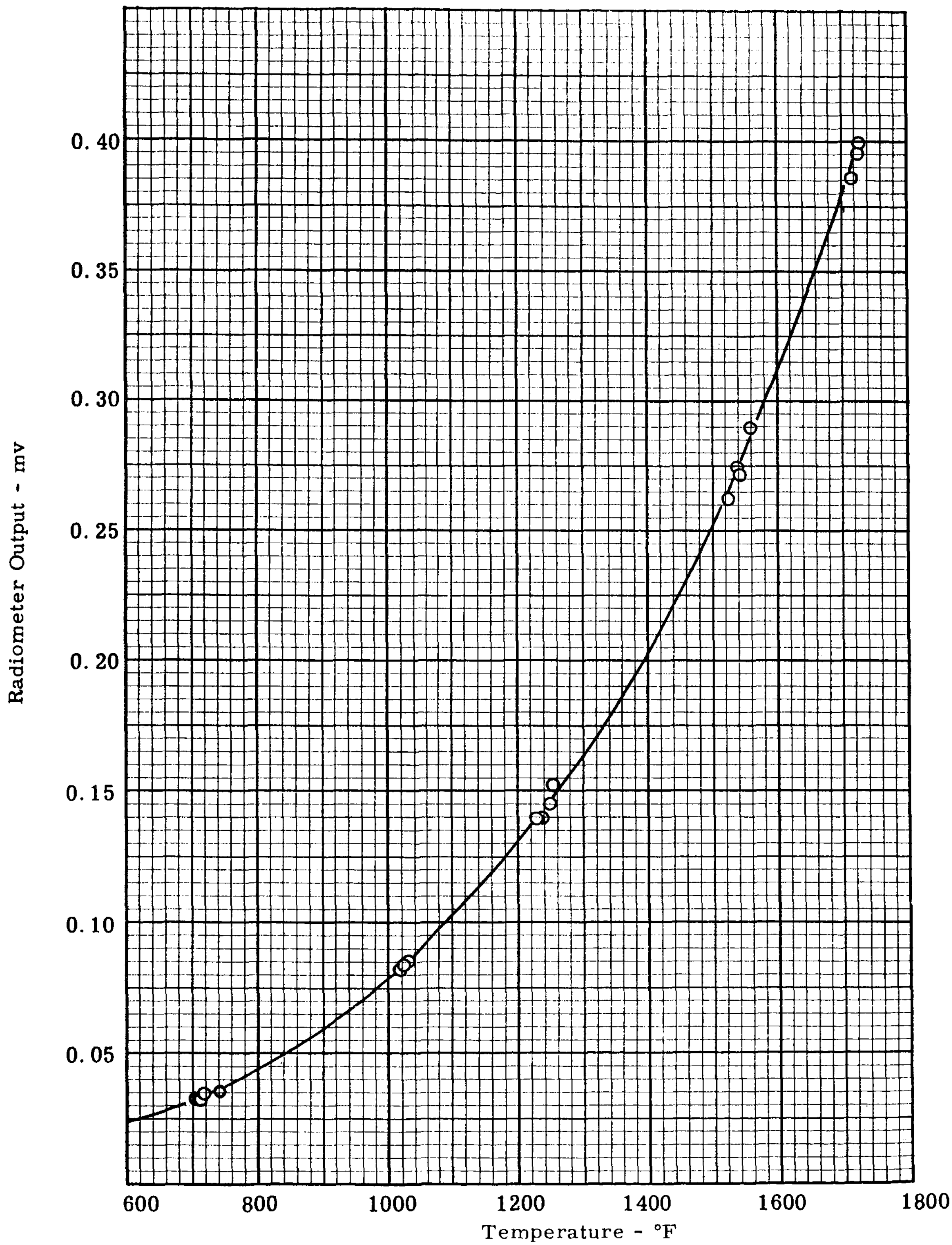


Figure 2. Recalibration of Radiometer Output vs Temperature for Blackbody Radiation in Standard Apparatus

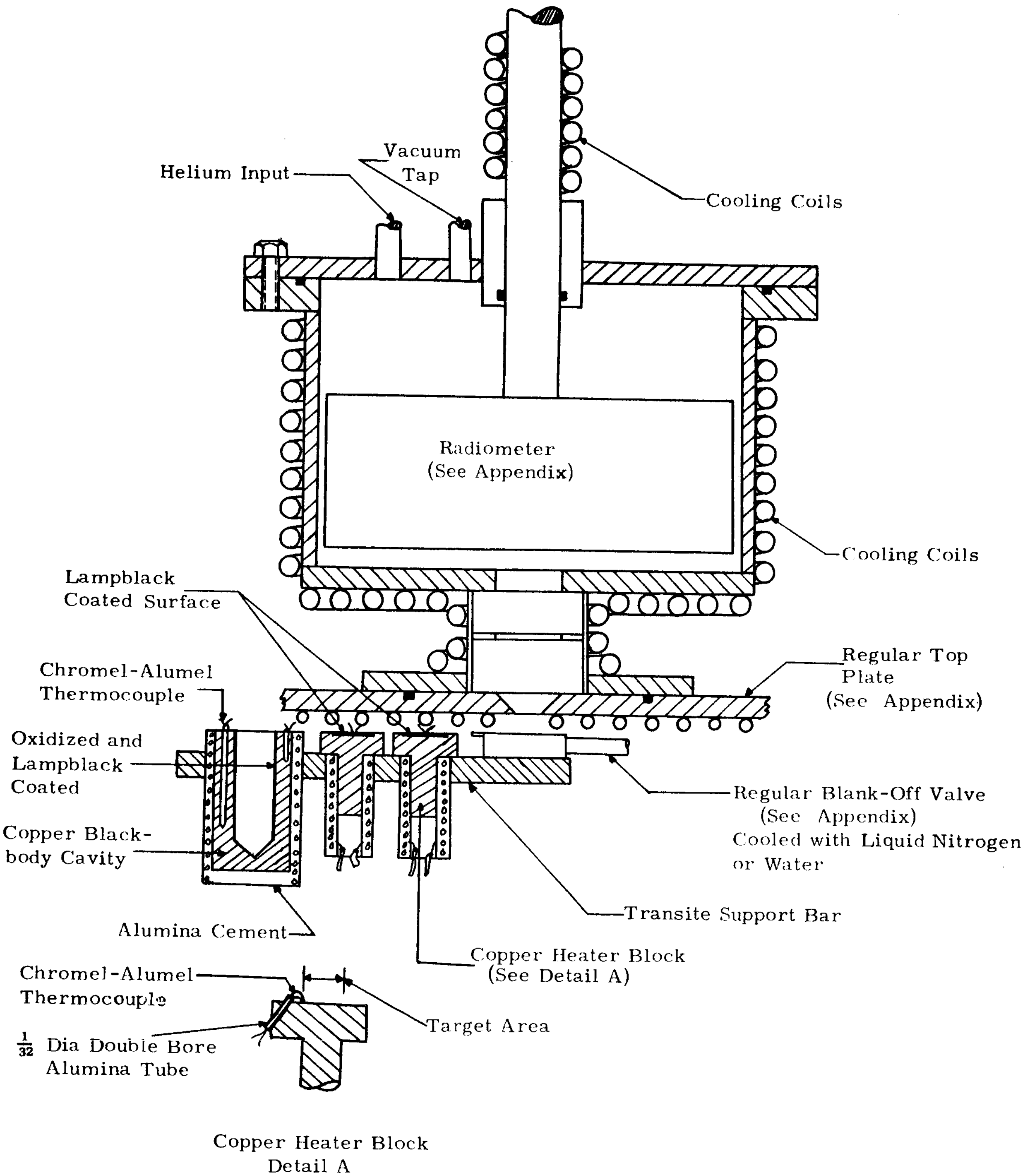
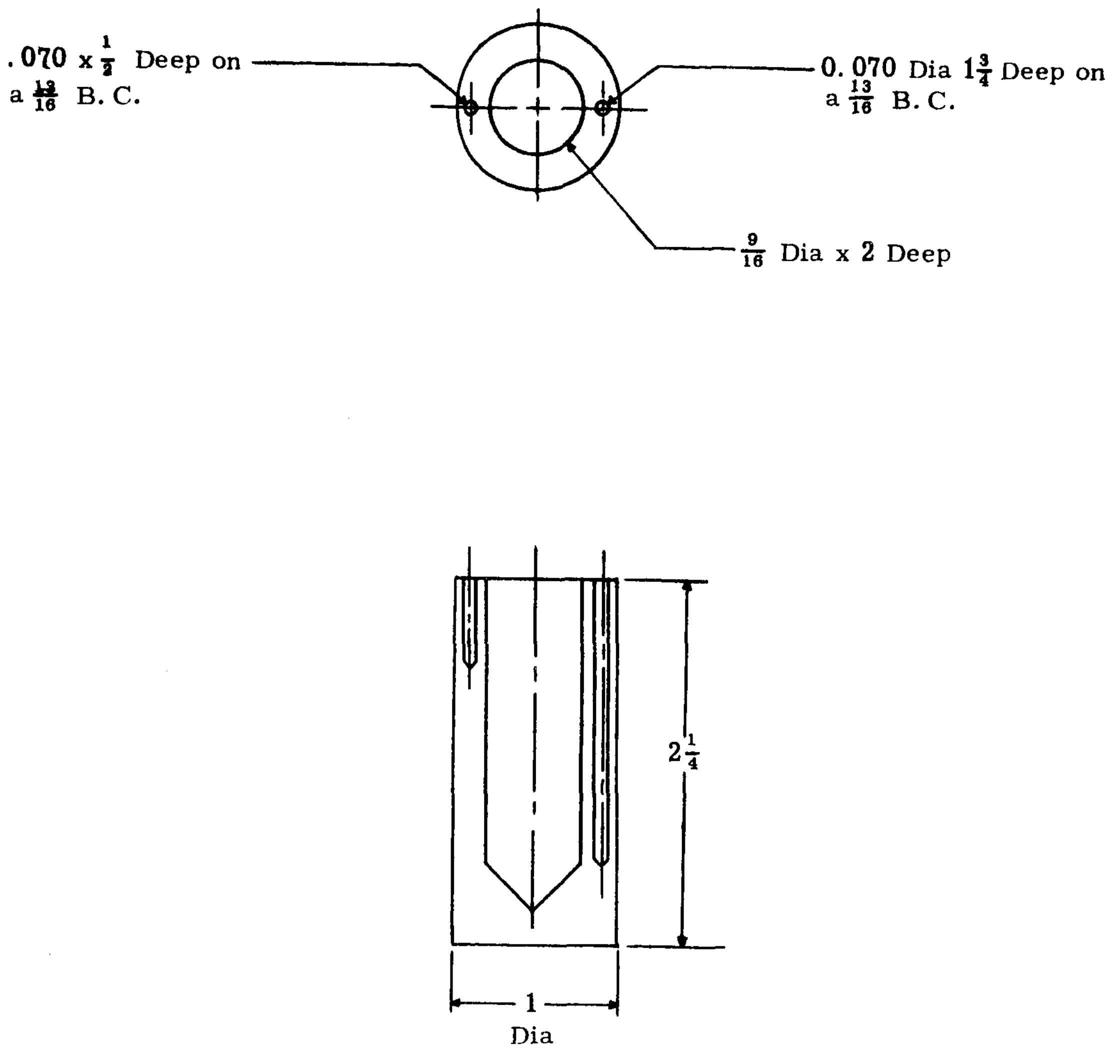


Figure 3. Modified Apparatus for Emittance Determination from 70°F to 800°F



Material-Copper

Figure 4. Blackbody Cavity Configuration for Modified Apparatus

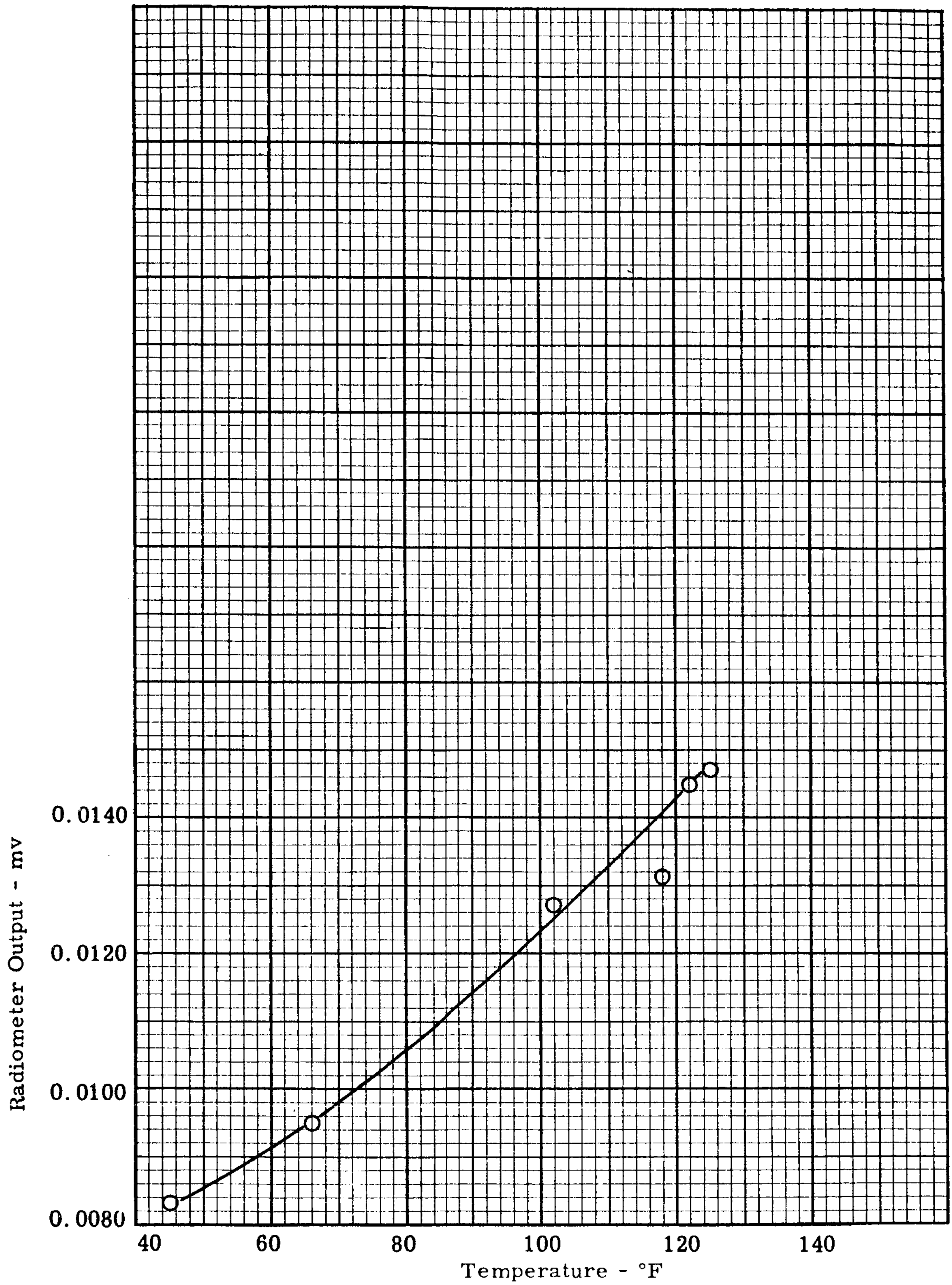


Figure 5. Liquid Nitrogen Cooled Radiometer Output vs Temperature for Blackbody Radiation in Modified Apparatus

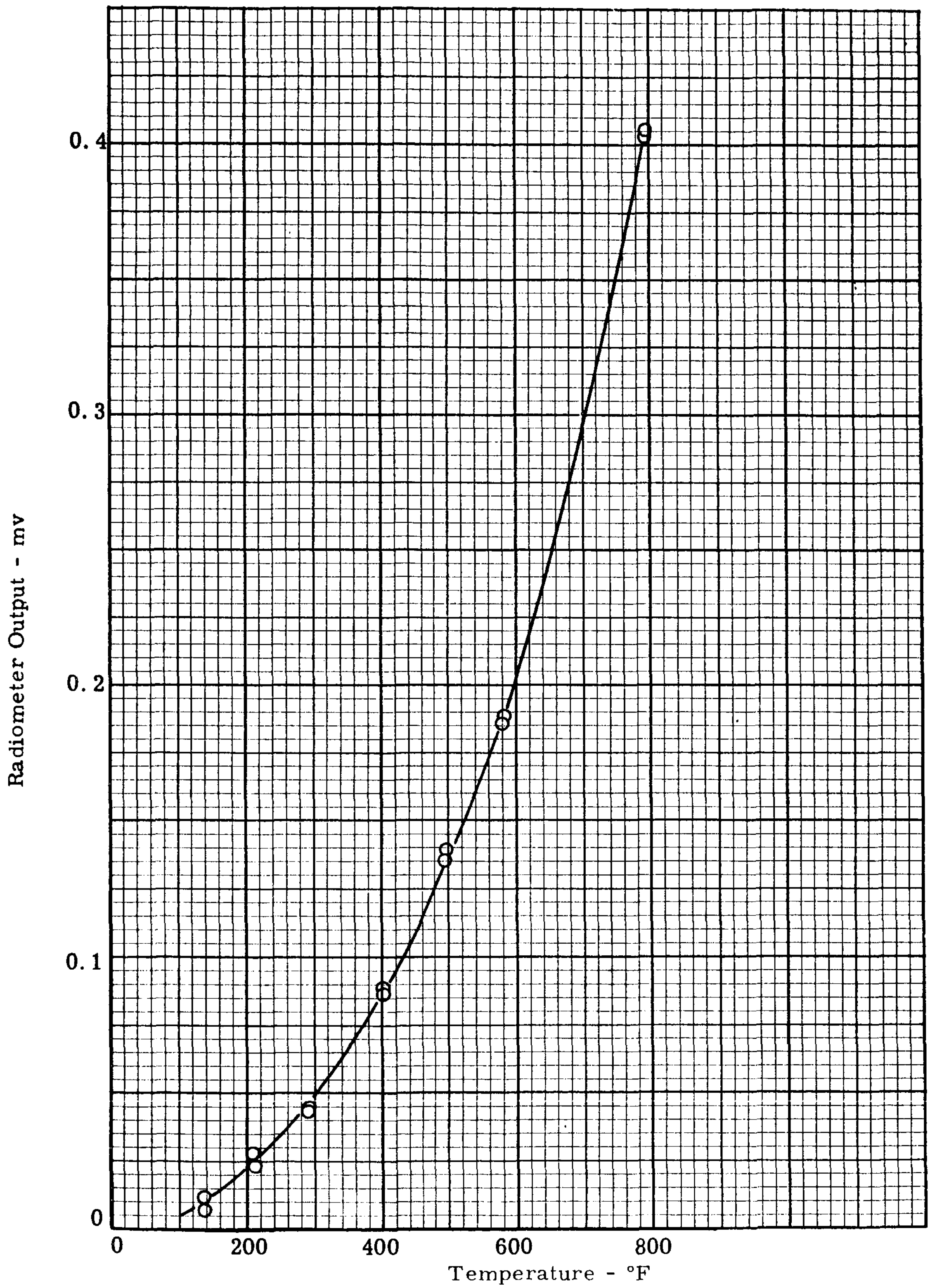


Figure 6. Water Cooled Radiometer Output vs Temperature for Blackbody Radiation in Modified Apparatus

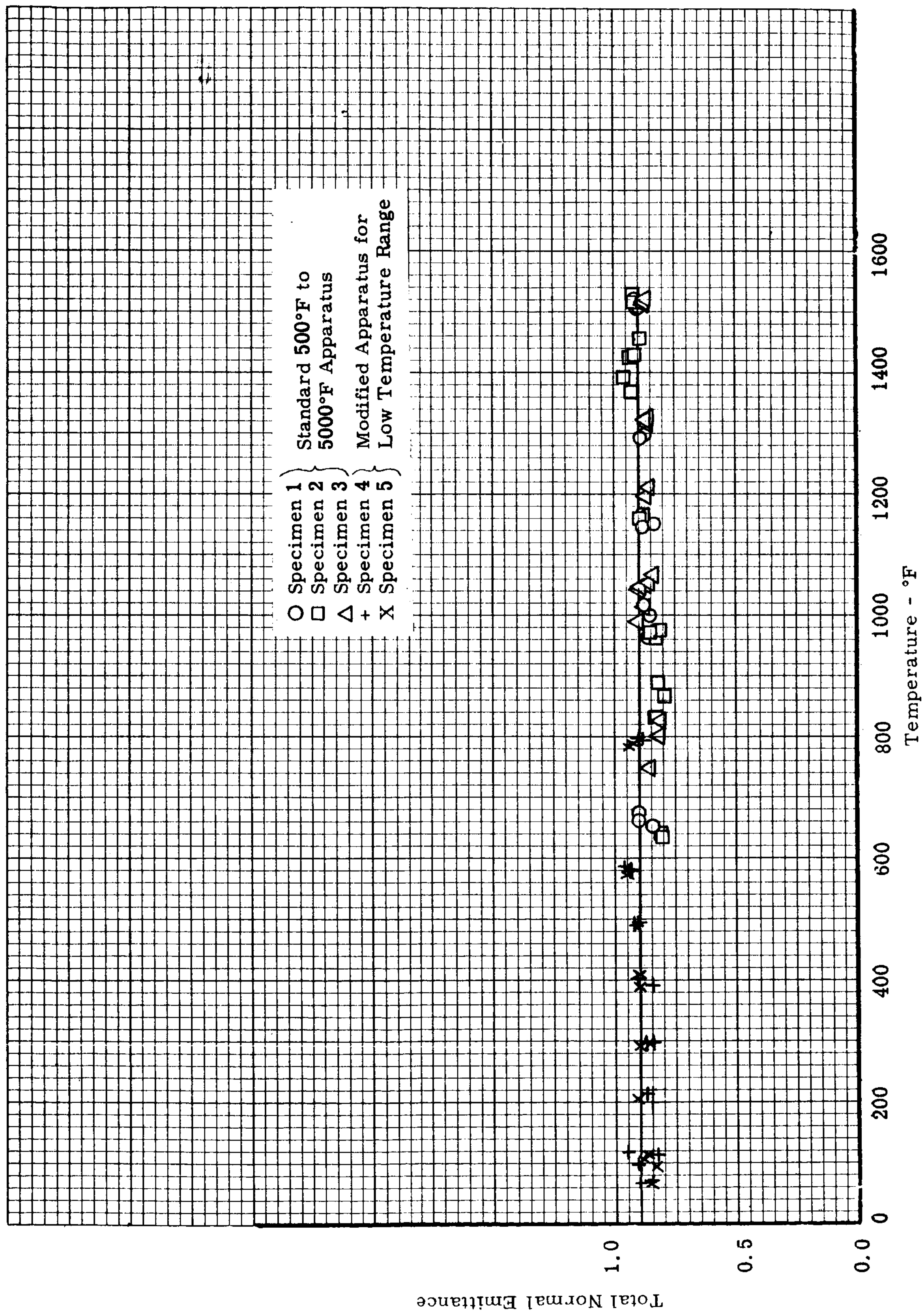


Figure 7. Total Normal Emittance of a Thin Lampblack Coating on a Smooth Copper Surface

Table 1

Total Normal Emittance of Lampblack Coating on a Smooth Copper Surface

Time	Specimen Temperature °F	Radiometer Output for Specimen MV	Radiometer Output for Blackbody MV	Total Normal Emittance
Standard 500°F to 5000°F Apparatus (Radiometer Water Cooled)				
Specimen 1				
1:50	661	0.026	0.029	0.90
1:54	658	0.024	0.028	0.86
1:59	671	0.027	0.030	0.90
2:30	983	0.065	0.075	0.87
2:35	1004	0.069	0.080	0.86
2:40	1021	0.073	0.083	0.88
2:48	1148	0.104	0.117	0.89
2:56	1152	0.102	0.118	0.86
3:02	1162	0.108	0.120	0.90
3:08	1297	0.146	0.163	0.90
3:13	1320	0.150	0.172	0.87
3:18	1300	0.144	0.164	0.87
3:31	1527	0.249	0.267	0.93
3:36	1520	0.244	0.263	0.93
3:41	1511	0.235	0.259	0.91
3:47	1510	0.240	0.259	0.93
Specimen 2				
1:30	646	0.023	0.028	0.82
1:47	837	0.041	0.049	0.84
1:52	870	0.044	0.054	0.81
1:56	889	0.047	0.057	0.82
2:04	977	0.060	0.073	0.82
2:07	963	0.060	0.071	0.84
2:10	970	0.062	0.072	0.86
2:18	1154	0.108	0.117	0.92
2:23	1145	0.104	0.115	0.91
2:28	1160	0.108	0.119	0.91
2:36	1374	0.181	0.193	0.94
2:42	1399	0.197	0.204	0.97
2:48	1437	0.203	0.220	0.92
2:52	1424	0.199	0.215	0.93
2:56	1460	0.209	0.232	0.90
3:12	1516	0.243	0.261	0.98
3:17	1533	0.248	0.271	0.92
(During and after above run, surface coating was observed as not uniform)				
Specimen 3				
1:40	748	0.032	0.037	0.86
1:46	804	0.037	0.045	0.82
1:51	828	0.039	0.048	0.81
1:57	989	0.069	0.076	0.91
2:03	1015	0.071	0.082	0.87
2:08	1043	0.078	0.088	0.89
2:12	1066	0.080	0.094	0.85
2:16	1048	0.080	0.088	0.91
2:20	1048	0.079	0.088	0.90
2:25	1049	0.077	0.090	0.86
2:30	1047	0.081	0.089	0.91
2:35	1039	0.078	0.087	0.90
2:50	1167	0.106	0.121	0.88
2:57	1197	0.113	0.130	0.87
3:02	1209	0.115	0.133	0.86
3:27	1329	0.151	0.175	0.86
3:33	1317	0.148	0.170	0.87
3:38	1320	0.149	0.172	0.87
3:53	1528	0.236	0.267	0.88
3:58	1505	0.229	0.255	0.89
4:04	1501	0.226	0.253	0.89

Table 1 (continued)

Total Normal Emittance of Lampblack Coating on a Smooth Copper Surface

Time	Specimen Temperature °F	Radiometer Output for Specimen MV	Radiometer Output for Blackbody MV	Total Normal Emittance
Modified Apparatus for Low Temperature Range (Radiometer Cooled with Liquid Nitrogen)				
Specimen 4				
10:42	68	.0082	.0097	.85
10:51	64	.0085	.0094	.90
11:03	99	.0111	.0122	.91
11:09	118	.0134	.0141	.95
11:25	113	.0119	.0136	.83
Specimen 5				
10:44	63	.0080	.0093	.86
11:00	93	.0098	.0117	.84
11:19	115	.0120	.0138	.87
11:28	108	.0116	.0131	.89
Modified Apparatus for Low Temperature Range (Radiometer Water Cooled)				
Specimen 4, Run1				
11:48	212	.0226	.026	.87
1:08	293	.0410	.048	.85
1:24	293	.0419	.048	.87
2:58	401	.0790	.087	.91
3:24	396	.0724	.086	.84
4:07	487	.1194	.130	.92
4:20	491	.1208	.132	.91
Run 2				
10:44	585	.1846	.191	.97
11:01	579	.1765	.187	.95
12:50	795	.3603	.404	.89
1:07	795	.3651	.404	.90
Specimen 5, Run 1				
11:52	204	.0219	.024	.91
1:11	291	.0425	.047	.90
1:27	290	.0414	.047	.88
3:01	386	.0740	.081	.91
3:28	410	.0816	.091	.90
Run 2				
10:48	570	.1772	.181	.98
11:05	568	.1721	.180	.96
12:55	784	.3706	.391	.95
1:12	792	.3736	.401	.93

APPENDIX

TOTAL NORMAL EMITTANCE TO 5000°F

TOTAL NORMAL EMITTANCE TO 5000° F

General

Emittance is measured by comparing the energy received by a radiometer from the sample to that received from a blackbody cavity maintained at the same temperature.

The equipment may be divided into three main parts: the induction heating furnace, the radiometer, and the temperature measurement equipment. Figure 1 shows a picture of the complete equipment.

Description of Apparatus

A cross section of the apparatus is shown in Figure 2. The specimen (1) is supported in the center of the flat concentrator induction coil (2) by a zirconia cylinder filled with fine zirconia grog and tungsten wires (3). The zirconia cylinder rests on a crucible filled with coarse zirconia grog (4). The radiometer (5) views the specimen from directly above through a water-cooled tube (6). A water-cooled optical valve (7) is used to blank off the specimen from the radiometer. Optical-temperature readings are taken through the main port (8), which may be pushed in to view the specimen through a mirror (9) from directly above. When radiometer readings are being taken, the main port is pulled out and away from the line of sight of the radiometer. Auxiliary port (10) is used to view the specimen directly as a check for the main port. Both viewing ports contain sapphire windows. The portion of the furnace above the specimen (11) is water-cooled to eliminate any possibility of energy being reflected back onto the specimen surface. The emittance furnace is built of steel and sealed with "O" rings so that a vacuum may be attained.

The radiometer, see Figure 3, was constructed according to Snyder[1] and Gier[2] with some modifications. The receiver element consists of approximately 160 turns of No. 40 AWG bare-constantan wire (104 turns

---

1 Snyder, N. W., Gier, J. T., and Dunkle, R. V., "Total Normal Emissivity Measurements on Aircraft Materials Between 100 and 1000° F," Trans. of the A. S. M. E., Vol. 77, 1944, p. 1011.

2 Gier, J. T., and Boelter, L. M. K., "The Silver-Constantan Plated Thermopile," Temperature - Its Measurement and Control in Science and Industry, American Institute of Physics, 1941, p. 1284.

per inch) wound around a plastic insulator strip about 2" long by $1\frac{5}{8}$" wide by $\frac{5}{16}$" thick. Silver was electroplated in several stages onto the constantan coil so that two $\frac{1}{8}$" wide lines of silver-constantan junctions, $\frac{1}{2}$" apart, were formed on the same side of the coil and across all of the wire turns. The remainder of the entire coil was silver plated. Each of the two lines of junctions was covered with a thin, narrow strip of black paper. One of these junction lines is designated as the active or "hot" junction and is placed to receive energy from the sample. The other is shielded and termed the passive or "cold" junction.

In order to shield the element from extraneous radiation, a cylindrical housing is placed immediately around the thermopile. The front of the housing contains a rectangular opening $\frac{1}{4}$" by $1\frac{1}{2}$" to allow the element to "see" the specimen. The actual limiting of the receiver field is accomplished by this rectangular slit and the $\frac{1}{4}$" round stop (12) just above the specimen. Additional stops in the water-cooled tube were installed as an added insurance to further minimize spurious reflections. The radiometer views the specimen directly. This eliminates the possibility of dirty lenses affecting the reading and, also, eliminates the spectral selectivity of the different types of materials used as windows.

The voltage generated by the receiver is measured with a Type K-3 Leeds and Northrup potentiometer in conjunction with an L and N Type 2430 DC galvanometer of 0.43 microvolts per millimeter deflection sensitivity. Temperatures are measured with a Leeds and Northrup portable potentiometer.

The receiver element was calibrated against a carbon-filament lamp of known radiation[3] and demonstrated a sensitivity of 8.66 Btu/hr/sq ft/millivolt.

The radiometer was checked, also, against an Eppley thermopile with 12 bismuth-silver junctions and a 1-mm quartz window and agreed within 10% scatter of data points. By factory calibration the sensitivity of the Eppley thermopile is 0.048 microvolts/microwatt/sq cm.

The optical pyrometers used are L and N catalog type 8622 calibrated in accordance with the International Critical Table of 1948 for an emittance of unity.

[3] Lamp No. C584, calibration by the National Bureau of Standards and reported in NBS Report 132737 A, July 1, 1952.

Calibration Procedure

To calibrate the radiometer for blackbody radiation, a blackbody cavity with a 6 to 1 aspect ratio made from graphite was used. The blackbody cavity was insulated by zirconia grog and lampblack placed in the annulus between the blackbody and the load coil, see Figure 4.

The accurate determinations of the specimen and blackbody temperatures are essential to good data. For the cavity-type blackbody, the temperatures are determined relatively easily by (1) thermocouples placed in the bottom of the cavity; (2) thermocouples dropped into the cavity; and (3) optical pyrometer observations. Up to 3000° F, agreement to within 15° F has been obtained regularly between these three readings. Above 3000° F the agreement between tungsten-rhenium couples and the optical pyrometer has been generally within 50° F or the repeatability of this type of thermocouple. Actually, the optical readings have no error other than those of the instrument calibration and the human error, which appears to provide a readout scatter of about 20° F at 4000° F.

Radiometer output versus temperature for blackbody radiation is plotted in Figure 5. Notice that the output is essentially linear from 2500° F to 5000° F with a slight curvature below 2000° F. As in house standards, the emittance of 304 stainless steel, tarnished tungsten, and graphite were measured, see Figure 6. The emittance of the stainless steel ranged from 0.15 at 700° F to 0.67 at 2000° F. These values are in close agreement with the literature values. The sanded CS graphite, also, checked out closely with the literature with values from 0.95 to 0.98.

Operating Procedures

The specimen is placed directly on the surface provided by the zirconia tube, grog, and tungsten wires. However, if the material of interest cannot be heated inductively, tungsten and tantalum heating discs are placed under the specimen with the specimen in contact with the tungsten disc.

The furnace is then evacuated to 15 mm of Hg and filled with high-purity, dry argon. This operation is carried out at least twice to assure an inert atmosphere. Throughout the run a slight pressure is maintained in the furnace by an argon purge, which is brought in through the radiometer enclosure and exhausted from the furnace housing, see Figure 2. In addition to maintaining an inert atmosphere, the purge flow tends to keep fumes away from the radiometer.

The temperature of the specimen is raised and maintained at the desired point by transferring energy to the specimen through the induction coil. About three hours are required to complete a single run with the temperature increasing stepwise but in uniform intervals. At each temperature level a radiometer reading is taken in conjunction with the temperature readings.

To obtain the radiometer reading, the following procedure is followed: As the specimen is heated, the blank-off valve is shut so that the thermopile can see no impulse. When the specimen temperature reaches steady state, a zero reading is obtained for the thermopile output. This reading is usually in the order of ± 0.02 millivolts. The blank-off valve is then opened, and the thermopile output increases several fold in a few seconds. The reading levels off as heat is transferred down the wires to the cold junction. The radiometer output is taken at the peak reading immediately after steady state. The net reading for that temperature is then obtained as the difference between the zero and steady-state reading.

If the blank-off valve were left open, the thermopile output would decrease slowly with time. After about 10 minutes, this reading might decrease by 50%; however, if the blank-off valve were shut and a new zero reading obtained, the difference between this new output and zero reading would be about the same as the original readings. The variation might be about 5 to 10%. The shift in readings is a result of the heating of the cold junctions.

The purge to the radiometer housing has no influence on the readings within the ranges at which the purge is operated. To determine this limit, the purge rate was increased to about 10 times the normal metered reading, and a small shift in readings of less than 1% was noted.

The temperature of the specimen is monitored by (1) thermocouples mounted directly on the target surface (usually held in place by a small zirconia pad) and (2) optical pyrometer readings on the target surface. Low temperature readings were made with thermocouples; however, in the intermediate temperature range from 1600° F to 2700° F a cross check was made between the thermocouple readings and the optical readings. The high-temperature measurements are made with an optical pyrometer. A main-port optical and an auxiliary-port optical-temperature reading are taken at each temperature level. The auxiliary-port temperature is normally used only as a check; however, if conditions warrant, such as a dirty main-port window or mirror, the auxiliary-port value may be used. Usually very good agreement is maintained between the main-port and auxiliary-port optical readings.

Emittance Calculation

The optical temperature readings must first be corrected to obtain true temperatures. The main-port reading is corrected for the sapphire window and mirror while the auxiliary-port reading is corrected for the sapphire window and the angle at which the port views the specimen. The corrections are shown as curves in Figure 7.

After assuming an arbitrary-initial, emittance value, the brightness temperature is corrected for this assumed emittance, see Figure 8. The blackbody output is then read at this "true" temperature from Figure 5. The ratio of the observed specimen radiometer output to the blackbody output is calculated and is the emittance of the material at that temperature. If the assumed emittance is correct, the calculated value will agree with it; if not, the calculated value must be used as the former assumed value and the process repeated until the assumed emittance value agrees with the calculated value. This iterative process will converge on the correct emittance value assuming graybody distribution of most of the energy at the particular temperature. The above process was programed for analysis by a digital computer.

Error Analysis

The above procedure for determining emittance is strictly correct only for those materials that radiate as graybodies, since the total emittance is assumed to be equal to the spectral emittance at the wavelength of the pyrometer. This approximation was used above to convert the brightness temperature to true temperature.

The error in emittance values for nongray materials will vary depending on the difference between the 0.665 microns spectral and the total emittance, and the distribution of radiant energy within the particular spectrum. If the deviation from graybody becomes very great at temperatures up to 2500° F, it is indicated by the thermocouple measurements. On materials of low emittance, such as tungsten, the emittance values calculated by this procedure could be in error by as much as 20% at the highest temperatures. However, it is believed that for most materials, the accuracy is within 10%. Several things indicate that the accuracy of the emittance values is good. First, the radiometer output versus temperature curves are orderly and almost linear with only normal data scatter. Second, the data obtained on two samples of the same material are in close agreement. Third, the values of emittance for the check samples agree very well with the literature, see Figure 6.

A statistical analysis of the data accuracy is of interest. Generally, the probable error in each blackbody reading is about 4%, and the probable error in each specimen reading is about 8%. If the data points are used to calculate emissivity, the maximum probable error would then be about 12%. The curve-fitting approach undoubtedly reduces this maximum to about 5%. As a general conclusion, the accuracy of the measuring system is well within the range of variation as is experienced by different finishes on the same material, the changing chemistry of the surface at the high temperatures, surface temperature measurements, and other variables.

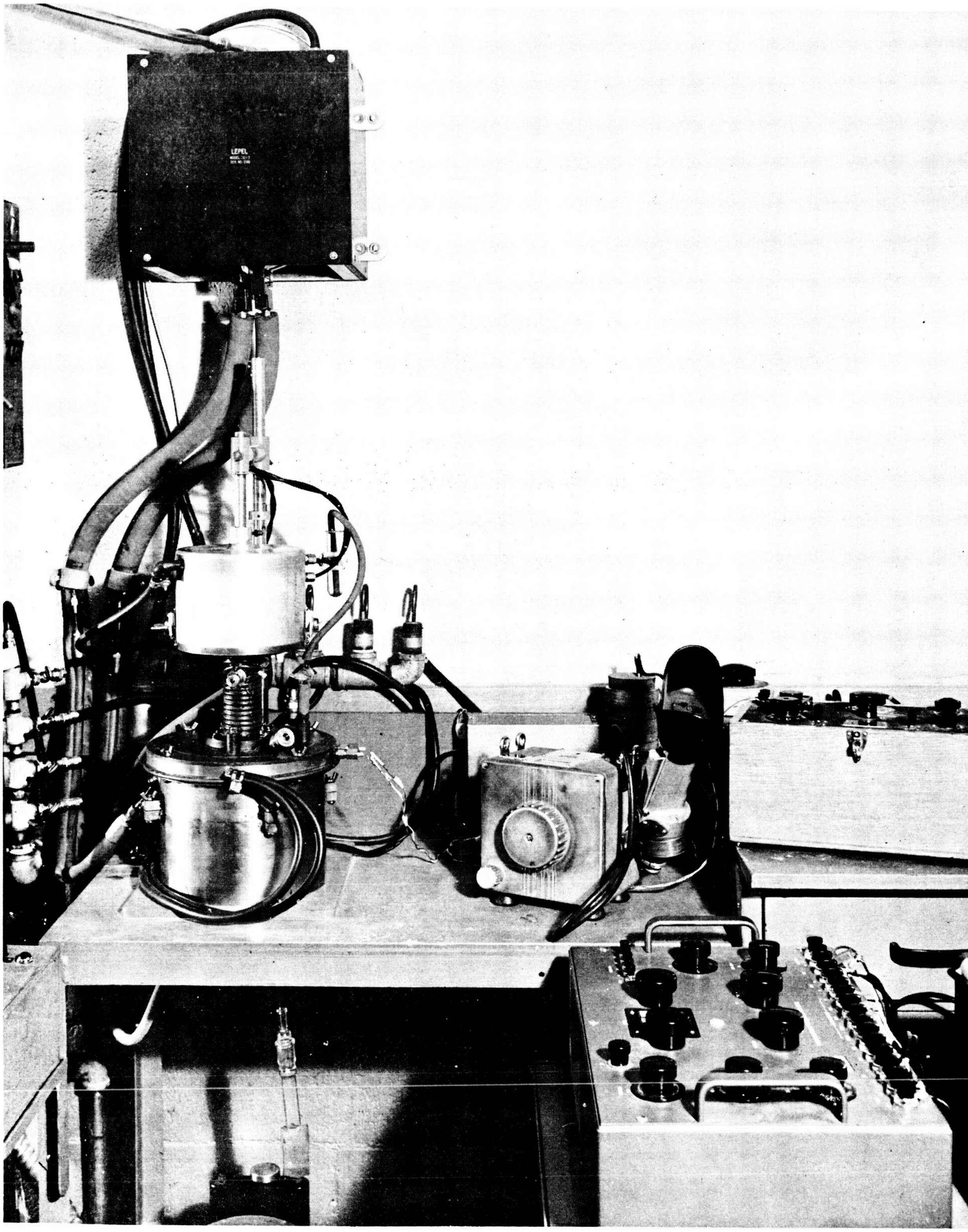


Figure 1. Picture of the Apparatus for Measuring Total Normal Emittance.

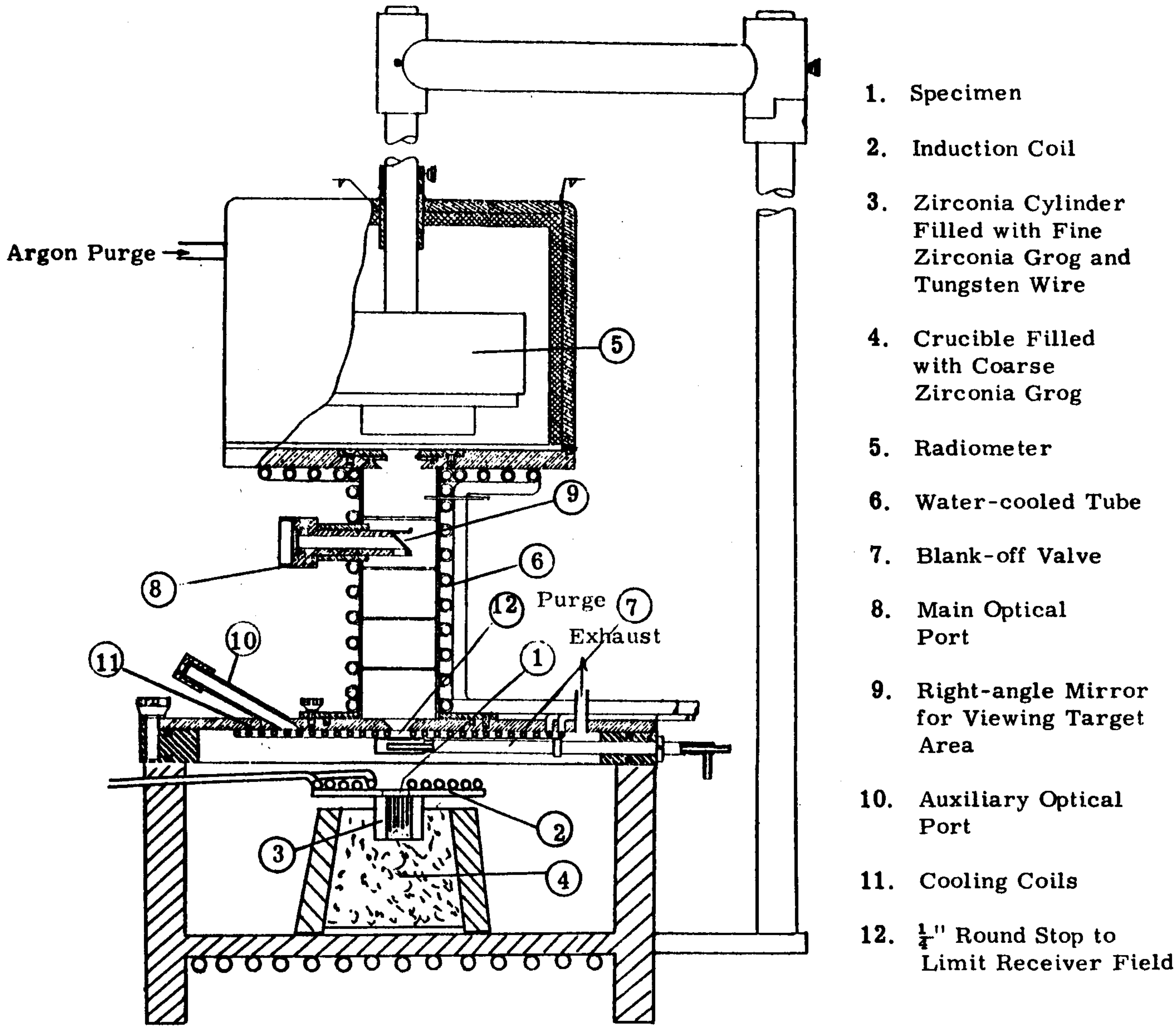


Figure 2. Cross Section of Emittance Apparatus with Flat Coil Furnace.

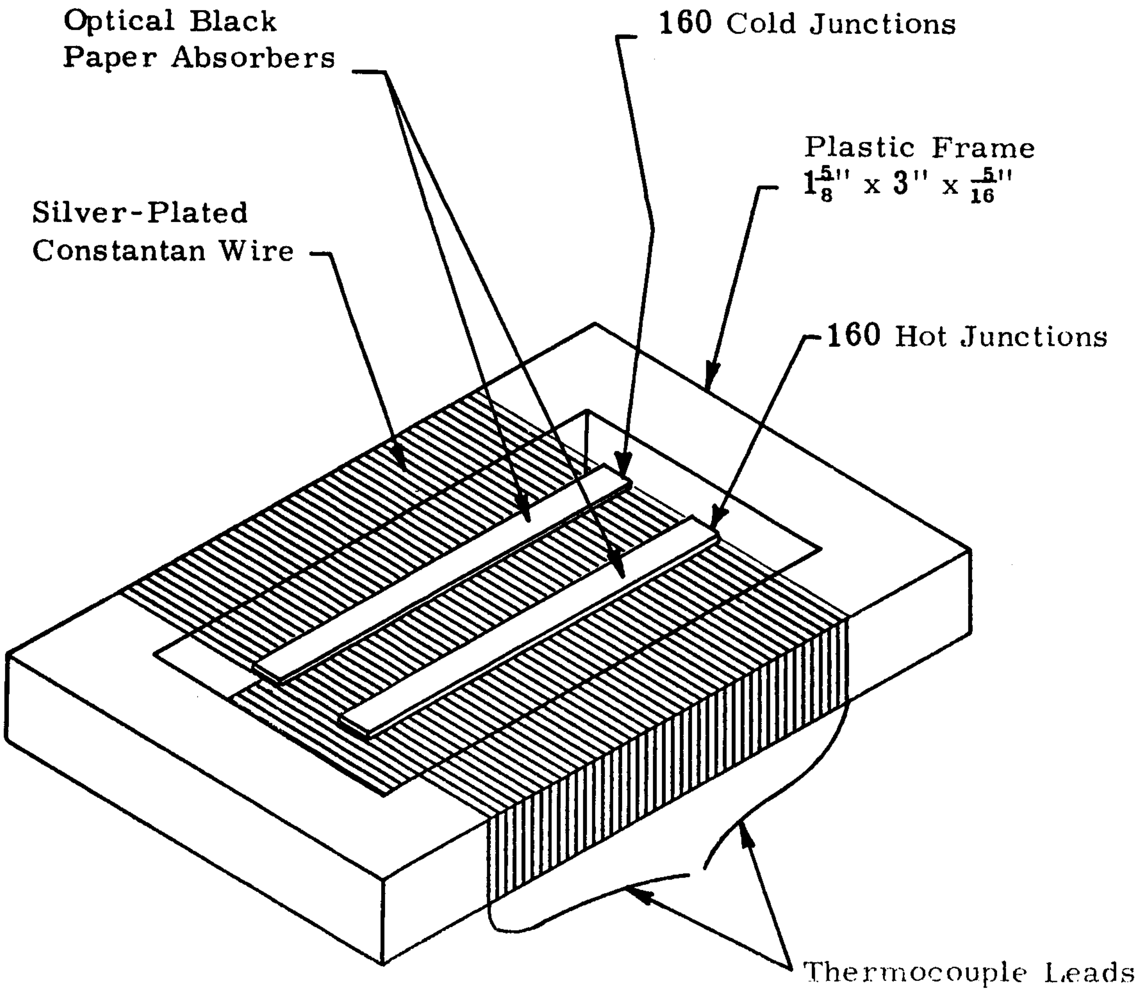


Figure 3. Schematic of 160-Junction Thermopile in Emittance Equipment.

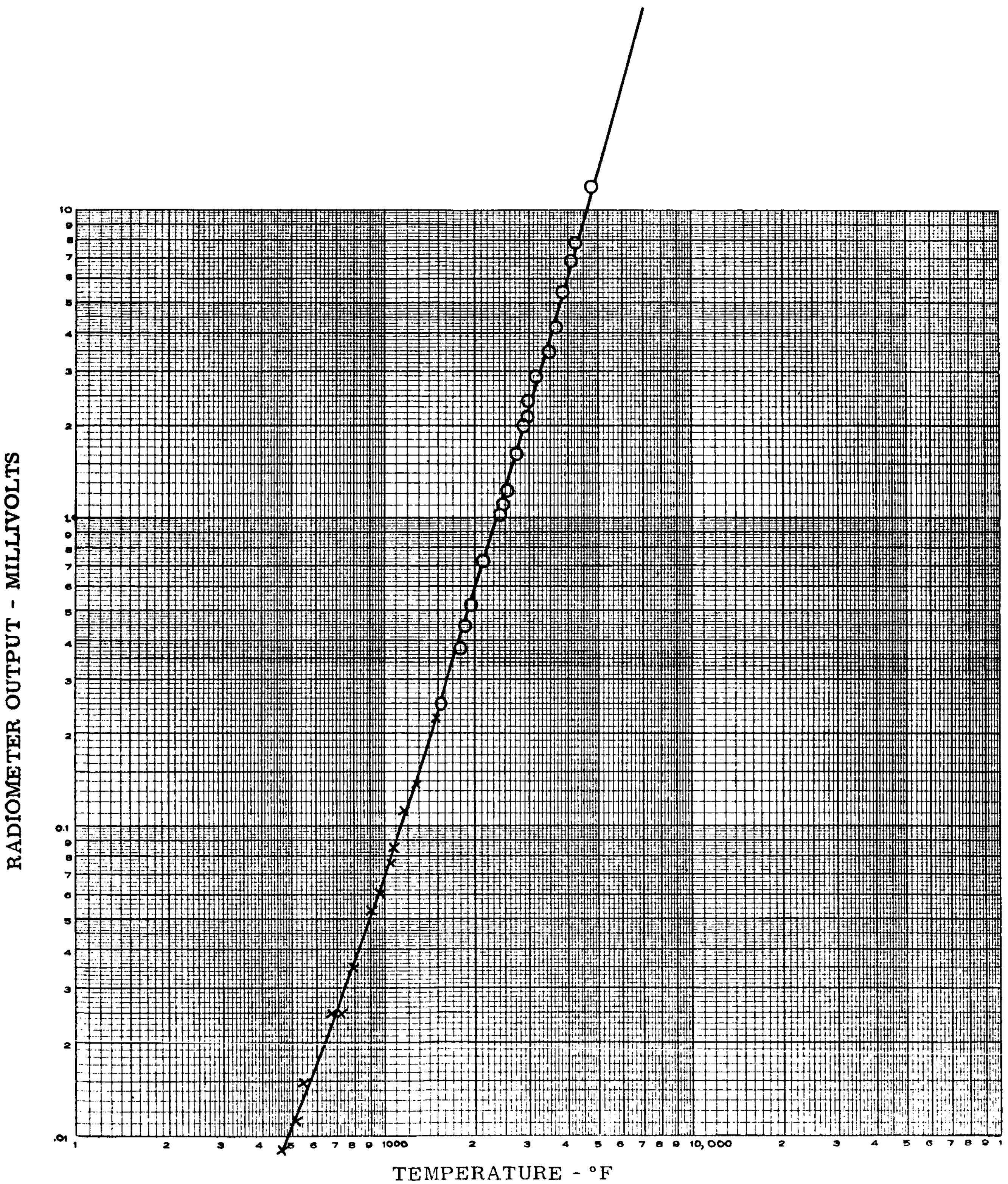


FIGURE 5 . RADIOMETER OUTPUT VERSUS TEMPERATURE FOR BLACK BODY RADIATION.

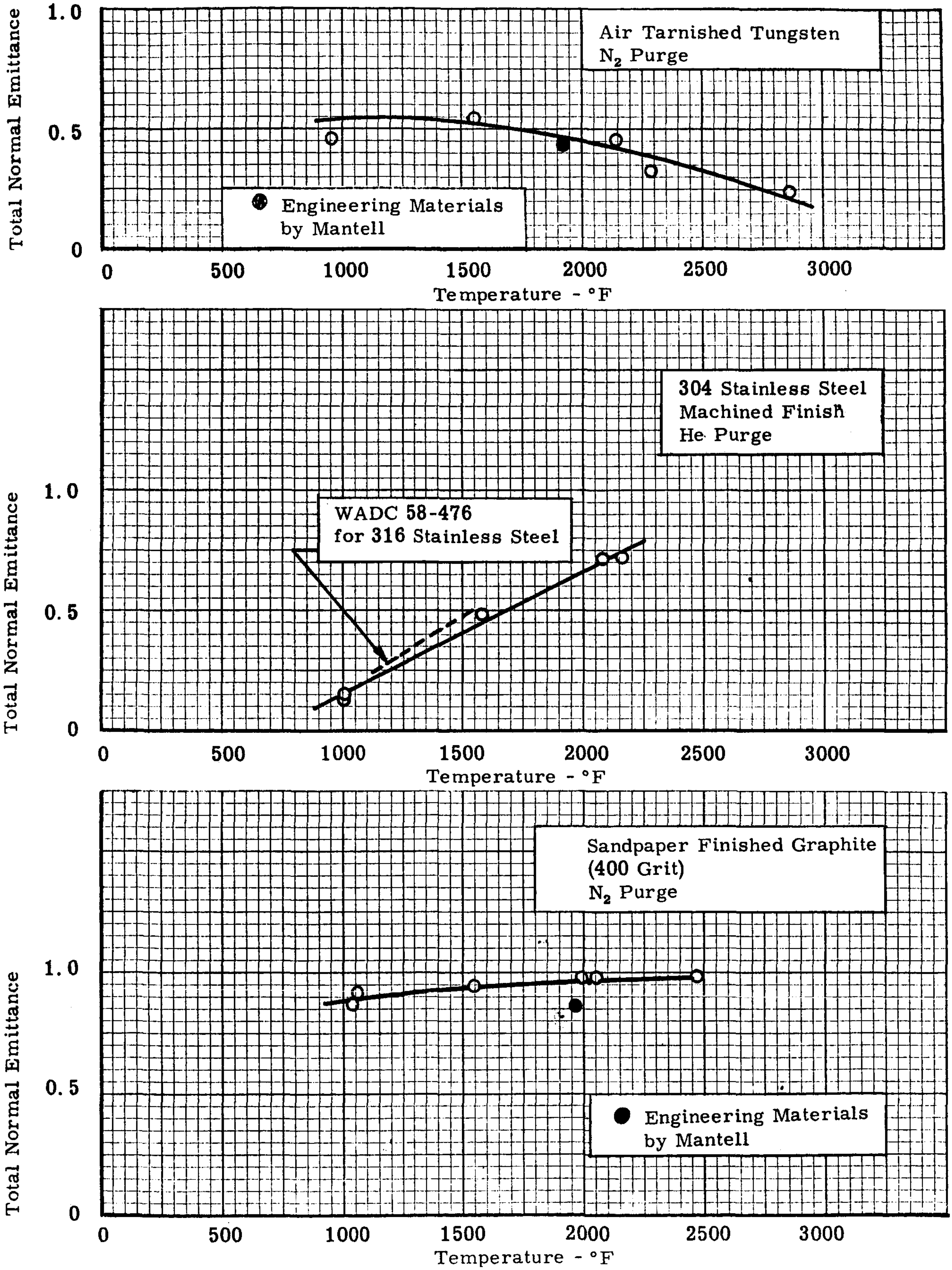


Figure 6. Calibration Standards for Total Normal Emittance.

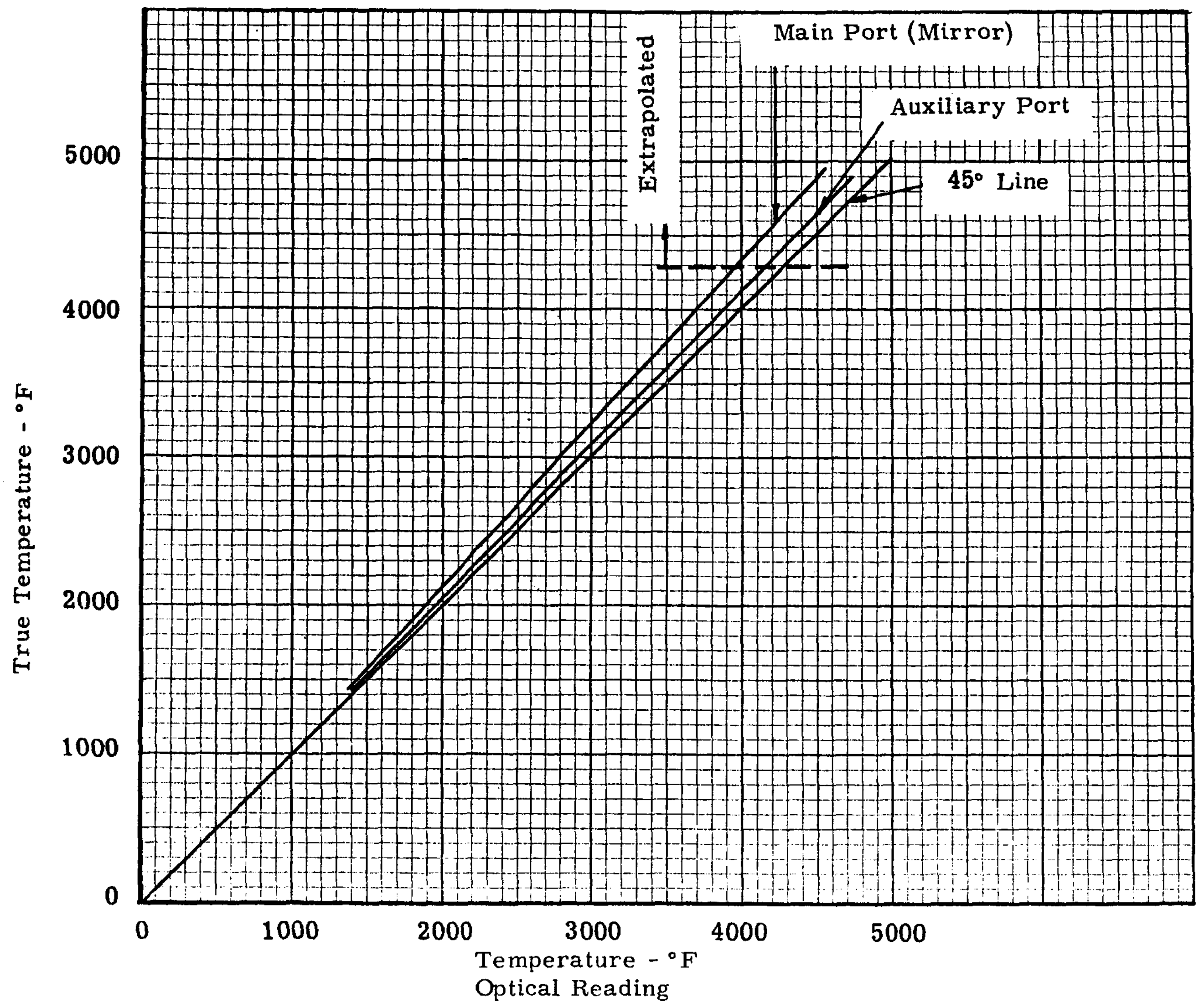


Figure 7 . Correction for Mirror and Sapphire Window in Emittance Apparatus.

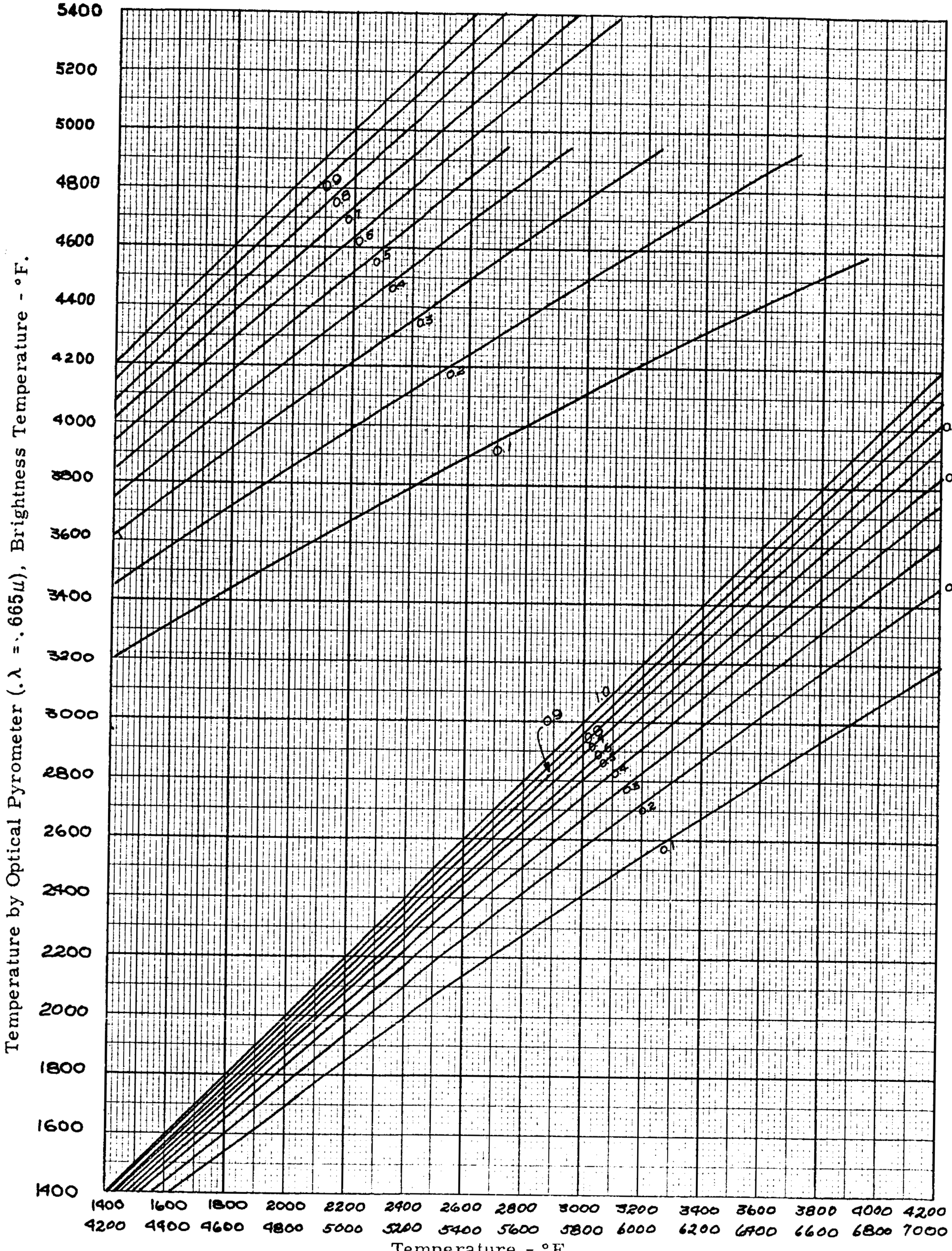


Figure 8. Correction for Brightness Temperature to True Temperature.